DEMONSTRATION AND EVALUATION OF THE AIR FORCE SITE CHARACTERIZATION AND ANALYSIS PENETROMETER SYSTEM IN SUPPORT OF NATURAL ATTENUATION INITIATIVES
VOLUME V - LIF SYSTEM OPERATION AND MAINTENANCE MANUAL

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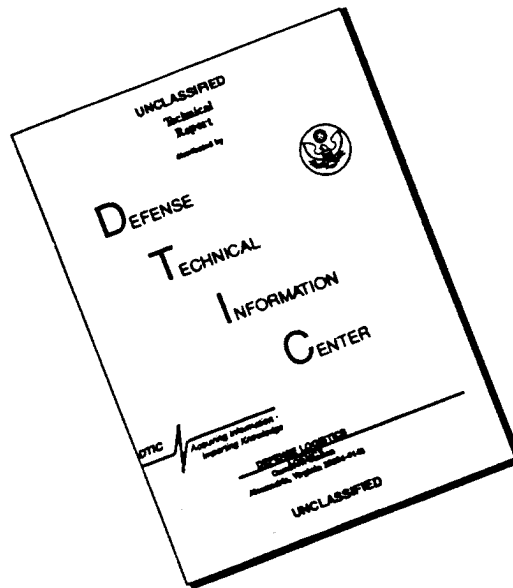Final Technical Report for Period October 1993 - December 1994

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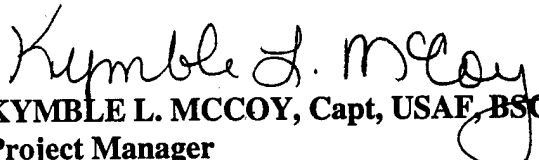
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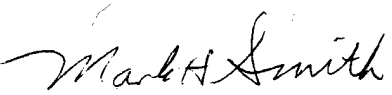
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This technical report has been reviewed by the Public Affairs Office (PA) and is releasable to the National Technical Information Service (NTIS), where it will be available to the general public, including foreign nationals.

This report has been reviewed and is approved for publication.

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A second-generation tunable Laser-Induced Fluorescence-Cone Penetrometer Test (LIF-CPT) system has been developed and demonstrated at three different Air Force Bases as an alternative site characterization technology. This represents an innovative technology for delineating soil contamination resulting from fuel spills. Applied Research Associates, Inc. and Dakota Technologies, Inc. jointly conducted the system development and demonstration project. Demonstrations consisted of 2-week efforts at each of Air Force Bases: Plattsburgh Patrick/Cape Canaveral, and Dover. The data collected during these demonstration supported both evaluation of the LIF-CPT systems along with support for selecting a site for a natural attenuation experiment the Air Force is planning. Data analysis indicates that the second generation system is operationally improved over the first-generation system and has improved detection capabilities. The improved detection capability is related to a new optical module used to focus laser light and filter the return signal to reduce the signal-to-noise ratio. Although the system is improved in many ways, some questions still exist concerning the influence soil type has had on some of the system responses. Further analysis is required to resolve these discrepancies.

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PREFACE

This report was prepared by Applied Research Associates, Inc. (ARA), 120-A Waterman Road, South Royalton, VT 05068 under U. S. Air Force Contract No. F08635-93-C-0020, SSG Subtask 8.01.1 for the Armstrong Laboratory Environics Directorate, AL/EQW-OL, 139 Barnes Dr. Suite 2, Building 1120, Tyndall AFB, FL 32403-5323.

This final report discusses the continued development of the combined technology of the cone penetrometer (CPT) and laser-induced fluorescence (LIF) as it pertains to the detection and quantification of petroleum oils and lubricants (POL's) within subsurface soils environments. Specifically the report covers a review of LIF-CPT technology; LIF-CPT system specifications; evaluation of the LIF-CPT probe under field conditions; and LIF data analysis/evaluation. The data and results of the field investigations were subsequently used to determine if bioventing or natural attenuation approaches are viable remedial alternatives at three U.S. Air Force Bases.

The work was performed between October 1993 and December 1994. The AL/EQW project officer was Mr. Bruce Nielsen.

EXECUTIVE SUMMARY

A. OBJECTIVE

Applied Research Inc. (ARA) and Dakota Technology, Inc. (DTI) were retained by the Armstrong Environics Directorate AL/EQ-QL to further develop laser-induced fluorescence-cone penetrometer technique (LIF-CPT) for use during environmental site investigations. The primary objective of the program was to develop and evaluate an improved LIF-CPT system for the characterization of fuel-contaminated sites. The evaluation was based on the results from demonstrations, testing and evaluation at three Air Force bases. A parallel goal of the demonstrations was to gather data for Engineering-Science, Inc. (E-S) to determine if bioventing or natural attenuation are viable alternatives for remediating numerous Air Force sites.

B. BACKGROUND

The Department of Defense (DoD) is seeking efficient and cost effective means to assess, remediate, and monitor petroleum-contaminated and hazardous waste sites at both active and decommissioned installations. The Air Force's Installation Restoration Program Information Management System (IRPIMS) database lists approximately 1,400 fuel-contaminated sites and 300 inactive firefighter training facilities; presently the IRPIMS database contains data from only one-half of the installations. Current environmental site investigations based on drilling technology are slow, expensive and potentially dangerous. Cone penetrometer (CPT) based investigations, on the other hand, allow real-time data collection and don't produce soil cuttings thus eliminating disposal costs and health and safety risks due to exposure.

Cone penetrometer testing gathers accurate in situ geotechnical information in a rapid and cost-effective manner. With adaptation of in situ geophysical and chemical sensors to the cone penetrometer probe, subsurface hydrogeology and the extent of contamination can be mapped simultaneously. The speed and continuous nature of the information generated by LIF-CPT reduces the need for costly and invasive subsurface sampling and installation of long term monitoring wells.

C. SCOPE

To fulfill the objectives of this project, the following tasks were completed; (1) Development, fabrication and integration of a field-deployable, wavelength-tunable LIF system, (2) Laboratory testing and evaluation of the LIF system, (3) Demonstration, testing and evaluation programs at Plattsburgh, Patrick/Cape Canaveral, and Dover Air Force Bases (AFB's), and (5) Delivery of a completed LIF system to the U. S. Army Corp. of Engineers.

Extensive evaluation and calibration of the LIF-CPT remained outside of the scope of this project. Such a study could not be completed during this project due to the dual objectives of

developing the LIF-CPT, and employing the tool and other CPT capabilities on a production basis for the engineering-cost evaluation conducted by E-S.

D. METHODOLOGY

The United State Army Corp of Engineers Waterways Experimental Station (USAE WES) initially developed the LIF-CPT using a mercury lamp as an excitation source downhole within the cone. The resulting fluorescence was collected and directed to a detection system located in the cone penetrometer truck via a single optical fiber. WES soon eliminated the mercury lamp in favor of a pulsed laser source external to the cone; a nitrogen laser system, limited to the emission of a single excitation wavelength of 337 nanometer (nm) was employed. This was useful for the detection of large multi-ring fuels such as Diesel Fuel Marine (DFM) but proved ineffective for "lighter" fuels such as jet fuels and gasoline which require excitation at shorter wavelength. Further research sponsored by the Air Force concluded that a tunable wavelength, pulsed laser (Nd:YAG) with a fiber optic probe and detection system would satisfy the needs of the Air Force. During the scope of the current program Applied Research Associates, Inc., in cooperation with Dakota Technology, Inc. (DTI), refined the Nd:YAG pulsed LIF-CPT system and demonstrated its utility in the field.

E. TEST DESCRIPTION

The test program consisted of two phases; (1) redesign and build a new laser system based on the findings of a previous LIF-CPT development program, and (2) evaluation of the LIF-CPT system under field conditions at three Air Force Bases. The redesign of the LIF-CPT system consisted of two major efforts: (1) optimizing the overall laser system performance by upgrading individual components with state-of-art components and repackaging the system improving portability and durability, (2) redesign the LIF-CPT probe to maximize the performance and durability and minimize cost. During the field demonstration and evaluation program several objectives were addressed. The primary technical focus was to evaluate the LIF-CPT system in the field for reliability, stability and repeatability, correlation of LIF-CPT intensity to contaminate concentration and evaluation of the sources of data scatter in the chemical and LIF-CPT data.

F. RESULTS

During the three demonstrations the laser system performed quite well. Many of the system improvements greatly enhanced both field utility and system stability. Minor improvements are still needed to enhance power stability, although the recording of the current power levels is highly beneficial.

Evaluation of the LIF response shows that there are effects related to both the soil type and moisture content of the materials being tested. The effect of soil type is fairly significant, since at some locations no LIF response was recorded in visibly contaminated clay samples. The effect of

moisture content is minimal. Further investigation into these responses would be highly beneficial. Finally, the limited data sets available permitted only speculation about possible correlations between LIF response and to be performed. The results form the statistical analysis are encouraging and additional testing performed.

G.   CONCLUSIONS

In general the LIF and chemical analytical data agree well qualitatively. Evaluation of the limited chemical and LIF data indicates that their may be a correlation between total BTEX and Xylene concentrations and LIF response. The background limit of the LIF response was determined to be independent of the soil type but may have a weak dependency on moisture content. The background limit of the current LIF-CPT probe configuration is approximately 50 counts. There are still some unanswered questions regarding the response of the LIF system in different soils. Areas known to be highly contaminated showed little or no response in fine grained soils (e.g., silts and clays). Insufficient chemical data was available to fully validate the LIF system.

H.   RECOMMENDATIONS

Additional data collection and evaluation is required to fully validate the LIF-CPT system. For future testing, it is strongly recommended that on-site analytical screening for Total Petroleum Hydrocarbons (TPH) by EPA method 418.1 be conducted on soil samples. This data can subsequently be used to correlate TPH concentration to LIF response. Combining objectives that include production-oriented data collection for other research is not recommended.

I.   APPLICATION

The LIF-CPT system can be implemented by the Air Force as the primary technology to conduct environmental site assessments where petroleum, oils and lubricants are the contaminants of interest. It could be used both as an initial screening tool and/or as a tool to monitor the effectiveness of a particular remedial effort.

J.   BENEFITS

Significant reductions in the time and cost of conducting environmental site assessments could be realized by implementing the LIF-CPT technology. This system provides superior data in real-time to use as a basis for selecting an appropriate remedial strategy.

K.   TRANSFERABILITY OF TECHNOLOGY

Virtually all industrial contractors involved with subsurface environmental site assessments where petroleum oils and lubricants are concerned could profit from the use of LIF-CPT technology. The industry in general is constantly seeking ways to conduct business faster, cheaper, and better; CPT-LIF fulfills these criteria.

TABLE OF CONTENTS

TABLE OF CONTENTS
(continued)

TABLE OF CONTENTS
(concluded)

LIST OF FIGURES

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SECTION I
INTRODUCTION

A. OBJECTIVE

The following is a general guide to operating the CS-3 system. The objective of this manual is to introduce the operator to the details necessary to successfully operate and maintain the CS-3 system. Once the operator is familiar with all of the aspects of operation, it is possible to change and add or delete some steps, based on experience and experimental design. It is up to the operator to become familiar with the commercial components which make up much of the instrument, such as the oscilloscope, computer, and Big-Sky laser, by reading the manufacturers' manuals. We suggest emphasis on the oscilloscope operation. **Please read and understand the entire manual before proceeding!**

B. GENERAL INFORMATION

The CS-3 system is a field-deployable instrument designed for use with a cone penetrometer test (CPT) system for the in situ screening of soil and groundwater for petroleum contamination. The instrument consists of two racks, one containing the laser source and detection systems, and the second containing the data analysis and data storage systems. The racks communicate via a 7.5-meter-long umbilical. Light is passed between the instrument and the soil which is probed via optical fibers which follow the path of the CPT electronics cable. Light delivery is through a sapphire window located in a custom "sub" which is fitted above the standard CPT cone.

The CS-3 instrument employs Laser-Induced Fluorescence (LIF) spectroscopy as the means of detection. Certain molecules, upon absorption of ultraviolet photons, will emit fluorescence light with a wavelength distribution unique to the molecule. The aromatic molecules targeted by the CS-3 LIF system are include BTEX and PAH's. Since most petroleum hydrocarbon substances contain a broad distribution of aromatic molecules, the emission spectrum (fluorescence wavelength distribution) of the contaminant is a combination of all excited molecules in the mixture. By varying the excitation wavelength (thus targeting different molecules in the mixture) and scanning the resultant emission spectrum, information about the type of contaminant can be gleaned.

The CS-3 system incorporates a tunable laser excitation source, which allows for the targeting of different fluorescent molecules in the contaminant. Light from the laser source is launched into an optical fiber and delivered to a window in the cone. The light is directed out of the window onto the soil or ground water. If contamination containing fluorescent molecules is present, the molecules will absorb the light and fluoresce. A small amount of the fluorescence passes back though the window and enters a collection optical fiber. The collected fluorescence is returned to the instrument via this fiber where it is directed into a monochromator for wavelength analysis. The monochromator separates the light into its various wavelength components and directs each component independently onto a photomultiplier tube (PMT). In the PMT, the light signal is converted into an electrical current. By scanning the wavelengths with the monochromator and measuring the resultant current at each wavelength, a spectrum of the fluorescence can be determined.

The laser source used in the CS-3 instrument is pulsed. The pulsed nature of the excitation allows for the determination of one additional dimension of information, the *lifetime* of the fluorescence. The lifetime of the fluorescence is effectively the average time interval for a given number of excited (similar) molecules to fluoresce. This lifetime can range from ~10 nsec (the lifetime of the laser pulse) to several hundred nsecs. Fluorescence lifetimes are characteristic of the type of molecule and its surroundings and are thus another indication of the contaminant composition.

Typically, the instrument is employed to record the fluorescence intensity as a function of the depth of the window below the ground surface. The resulting log is known as a fluorescence versus depth (FVD) profile. FVDs require choosing a particular fluorescence wavelength and monitoring the fluorescence intensity at this wavelength as the cone/window is pushed into the ground. The fluorescence monitoring wavelength is chosen based on the types of contaminants expected at the site. During a push, the signal from successive laser shots are averaged for one second (approximately 50 laser shots). The ASTM approved push rate is 2 cm/ second. Thus, an FVD typically has 2 cm vertical resolution. FVD information is used to delineate the vertical extent of contamination at a given horizontal location.

In addition to providing FVD information, the CS-3 instrument can differentiate petroleum contaminant types. This is accomplished by scanning the fluorescence emission wavelengths while the cone is stationary. By recording both the fluorescence intensity and fluorescence lifetime at each emission wavelength, a wavelength-time matrix (WTM) can be constructed. When viewed

in 3 dimensions, a WTM yields a "fingerprint" of the contaminant. This technique is particularly useful if multiple contamination sources are present on one site.

C. SCOPE

The CS-3 system and control program are improved versions of the CS-1 and CS-2 instruments developed at North Dakota State University (NDSU). The CS-3 software is written in Microsoft® Basic Professional 7.1 and is not a fully debugged program ready for commercial application. Program changes were made during field demonstrations when serious bugs and error-prone sections of the program were detected. Changes in the code will be necessary if this system is to be integrated into any other CPT systems other than the one used in the demonstration because of the CPT truck/CS-3 interface.

SECTION II
LASER SAFETY

A. DANGER: INVISIBLE LASER RADIATION

The operator of this instrument must be aware of the danger involved in working with UV and high power visible lasers. The purpose of this section is to inform the operator of these dangers and summarize safe operating practices. It is the responsibility of the operator to ensure that all personnel are adequately protected from exposure to the lasers during operation of the CS-3 instrument.

During normal (cover on) operation, with the optical fiber connected to the output of the instrument, the laser output is limited to the cone. Energy levels emitted from the cone fall below hazardous levels 1 foot from the window. One should avoid prolonged skin exposure to the UV. Wear gloves when cleaning the window or placing reference solutions on the cone. Although the beam emitted from the cone is not a visible danger, eye protection should be worn. Plastic goggles often act as excellent eye protection from the UV output of the instrument. To determine if the safety goggles required at most sites are ample protection against the UV output of the instrument, check a fluorescent card for fluorescence after the beam has passed through the lens of the goggles.

Operation of the instrument with the excitation fiber removed from the back panel will result in hazardous UV output from the output feedthrough. Avoid exposing the skin to this radiation. Ensure that the instrument is situated in such a way that no one can look into this aperture while the laser is running. Ensure that all personnel in the area have adequate (clear plastic) eye protection. Be aware that the UV output is invisible to the human eye. Use a fluorescent card to locate the beam (most business cards work well). Ensure that the laser is OFF (use the shutter) when connecting and disconnecting the fiber cables.

Operation of the instrument with the cover removed from the optical breadboard is extremely dangerous. Under no circumstances should unauthorized or untrained personnel be allowed to operate or view the system with the cover removed. *The cover should only be removed when the laser is off.* An interlock is in place to ensure that the system will not run with the cover off.

With the lid removed, dangerous levels of both visible and UV light are present. The laser goggles supplied with the system are adequate protection only against exposure to the 532 nm output of the YAG laser. They are not designed for eye protection in the yellow (560 nm - 600 nm) region of the visible spectrum. Eye damage can take place in one shot of the laser, even from a small reflection of the beam. Ensure that all of the safety practices outlined below are followed if the laser is operated with the cover removed from the optical breadboard.

B. PRECAUTIONS FOR SAFE LASER OPERATION

1. Use protective eyewear at all times when using the laser system. The eyewear should be rated to protect you from the above wavelengths. Consult ANSI, ACGIH, or OSHA for guidance on goggles and safety matters.

2. Avoid looking directly at the laser output beam or reflections of the beam.

3. Do not wear reflective jewelry while manipulating laser optics.

4. Operate at the lowest possible intensity. When aligning or trouble shooting, begin with very low power in order to locate stray beams. Ensure that all stray beams are blocked before increasing the power.

5. Avoid blocking the beam with any part of your body.

6. Establish a controlled access area when working on the laser optics.

7. Maintain high ambient light levels so eye pupils remain constricted.

8. Post prominent signs near the laser operating area.

9. Leave the protective cover in place except when servicing the laser.

The safety precautions in the YAG laser manual should be read thoroughly. The top on the optical breadboard is ample protection from any light being generated by the YAG. If, for some reason, the top is removed while the laser is running, all precautions outlined for the YAG laser must be taken. The most important of these is wearing eye protection. Goggles for this purpose were ordered when the system was built and should be provided. Ensure that everyone working in the vicinity of the laser is warned of the danger and is informed of the precautions before operating the LIF system with the cover removed. Remember, these goggles are NOT adequate protection from the output of the dye laser. The operator is responsible for laser hygiene

while the cover is removed. Ensure that all personnel in the area are aware of the hazardous nature of the beams.

Ultraviolet light is invisible to the human eye. Always check the following areas with a fluorescent card (most business cards will work) to ensure that the UV beam generated by the LIF system is not being directed toward people - the bulkhead connector on the back of the optical breadboard and the exit end of the fiber probe.

Adequate eye protection from UV light is often supplied by eye glasses or simple safety goggles. To check if the glasses or goggles you choose will work, contact a laser safety hygienist. The optical fiber probe attaches to the LIF system on the aluminum bulkhead connector at the back of the optical breadboard. When no fiber is attached, UV light will be directed out the connector. Be sure that this beam is adequately blocked if the system is operated without a probe, for example during installation.

Operators using LIF cones should take precautions to avoid looking directly into the window while the laser is running. The safety glasses required for standard eye protection in a CPT vehicle are probably adequate protection from the UV beam, but this should be verified using the procedure outlined above. Note that the beam exiting the optical fiber expands quite rapidly. As a result, intensity levels ~ one foot from the cone window are below dangerous levels. This distance should be maintained at all times while the laser is operating. For implementations in which the beam is focused outside the cone window, the safe operating distance may be greater.

SECTION III
COMPONENT DESCRIPTIONS

The CS-3 system is composed of two racks with a separate computer monitor. As shown in Figure 1, Rack A is the electronics cabinet where the LIF data are analyzed and stored. Rack B is the laser cabinet where the excitation light is generated and fluorescence signal is collected. The following component descriptions will assist the operator in identifying the components of the CS-3 system. All component locations refer to Figure 1.

A. RACK A - ELECTRONICS RACK

1. Control Computer
(Items R, P, & O)

The CS-3 system is controlled by a 33 MHz 486DX computer from Gateway 2000®, (Item R), configured with 8 MB of RAM, an internal 250 MB Jumbo® tape backup system, a 3.5 inch diskette and a 424 MB IDE hard drive. The computer is equipped with a 14 inch non-interlaced monitor, (Item O), and a standard AT compatible keyboard (Item P). The computer controls the stepper motor controller and SPEX 270M via RS232 serial communication. Communication with the digital storage oscilloscope is achieved with a National Instruments® GPIB interface. A digital I/O board internal to the computer is used as a parallel interface to obtain depth information from the CPT truck. The LIF system is completely controlled via custom software run on the control computer.

2. Digital Storage Oscilloscope (DSO)
(Item S)

The Tektronix® TDS 620A is a digital storage oscilloscope capable of digitizing incoming waveforms at 2 gigasamples/second and is equipped with a GPIB interface. The DSO functions as the data capture system on the CS-3. Both the fluorescence signal from the PMT and the output energy of the UV beam (via an energy meter) can be monitored by the DSO. Control of the DSO is accomplished either via the front panel directly or, during an experiment, by the control computer.

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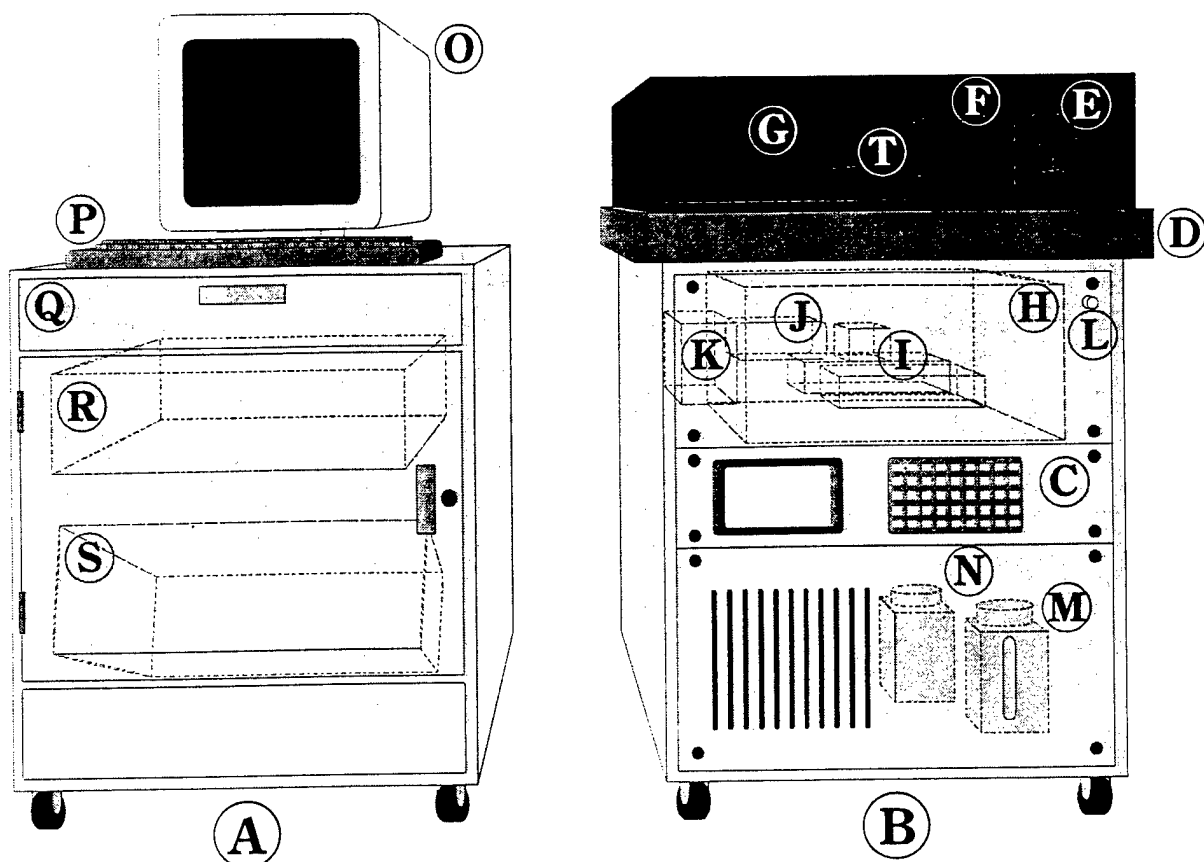
A. Electronics cabinet
B. Laser cabinet
C. Nd:YAG power supply
D. Optical breadboard
E. Frequency doubling crystal
F. Nd:YAG laser head
G. Dye laser
H. Monochromator
I. Stepper motor controller/driver
J. Photomultiplier tube and housing

K. High voltage power supply
L. Stepper motor reset button
M. Water reservoir
N. Dye reservoir
O. Monitor
P. Keyboard
Q. Utility Drawer
R. Computer
S. Oscilloscope
T. Temperature Controller

Figure 1. Component Level Diagram of the CS-3 System.

8

B. RACK B: LASER RACK

1. Nd:YAG Laser
(Items C & F)

The Nd:YAG laser consists of three parts interconnected via a wiring and coolant harness. The laser head (Item F) supplies the light at 532 nm (green) which drives the rest of the system. Power for the laser head is supplied by the pulse forming network (PFN), (Item C), which essentially discharges a capacitor through a lamp in the laser head. Finally, cooling of the laser head is accomplished using a water circulator, (Item M), located in the pump drawer. Information on the YAG laser is available in the manual supplied with the CS-3 system. *The YAG manual should be read and understood thoroughly before operating the CS-3 system.*

2. Dye Laser
(Item G)

The dye laser is located on the front one third of the optical breadboard and consists of two distinct segments. The oscillator segment (Item G) consists of a Bethune cell, rotation stage, grating, feedback mirror, and feedback wedge. Laser output from the oscillator is continuously tunable from 560 nm to 600 nm. The second segment of the dye laser is an amplifier cell which consists of a single Bethune cell located in front of the YAG laser head (Item F). The laser dye is circulated via a pump (Item N) located in the pump drawer.

3. Monochromator
(Item H)

A SPEX® 270M monochromator is located on the top shelf of the laser cabinet. It is a 0.27m monochromator equipped with a front entrance slit and a side exit slit. The monochromator is used to select the wavelength at which the fluorescence signal will be monitored. Both the slit width and the wavelength are selectable from the control software.

4. Photomultiplier Tube and Housing
(Items J)

A Hamamatsu® R928 photomultiplier tube (side-on) is housed in a light tight Products for Research® housing (Item J) connected to the side exit slit on the

9

monochromator. The system converts photons passing through the monochromator to an electrical current. The current pulse is converted to a voltage pulse directly on the housing using a 50 ohm terminating resistor.

5. High-Voltage Power Supply
(Item K)

The high-voltage power supply which supplies the PMT is a silver box located on the rear left hand side (facing the front of the cabinet) of the top shelf of the laser cabinet. In addition to the high voltage (HV) output, an HV/11 output is available to monitor the actual voltage supplied. (Note that most multimeters are limited to measuring voltages below those supplied by this supply.)

6. Frequency Doubling Crystal
(Item E)

The frequency doubling crystal is an angle tuned KDP crystal mounted on a motorized rotation stage. The crystal is enclosed in a sealed chamber to reduce exposure to humidity. The crystal is held at a constant temperature of 40°C by the crystal heater controller (Item T) to attenuate the effects ambient temperature changes. The heater controller is self contained. Any adjustments must be made by resetting the target set point.

7. Stepper Motor Controller
(Item I)

A Motion Control® stepper motor controller and driver system serves as the control system for the stepper motors located on each of the rotation stages on the breadboard. The stepper motor control the positioning of the rotation stages. The small black box houses the microprocessor and the two cigarette carton-sized boxes (stepper drivers) supply the motors with a current pulse train to move them. Stepper position is controlled with the control computer via RS232 serial interface. The wiring harness connecting the stepper power supplies to the stepper motors on the breadboard is routed through the two large black circular plastic connectors (CPC) on the back panel. *It is important that the motors not be disconnected from the stepper drivers when power is supplied to them! A 30-second wait is required before disconnecting the motors after powering down. This includes the two CPC connectors on the back of the optical breadboard. Permanent damage will result if they are disconnected while the power is on..*

SECTION IV
OPERATING SPECIFICATIONS

A. POWER REQUIREMENTS

Supply Voltage:	110VAC ± 10%, 60Hz
Supply Current:	Laser Rack - 30 Amp
	Electronics Rack - 20 Amp

B. FLUID REQUIREMENTS

YAG coolant:	Distilled Water
Dye circulator:	1×10^{-4} M Rhodamine 590 tetrafluoroborate in Methanol
Dye Solvent:	HPLC grade methanol

C. DOUBLING CRYSTAL REQUIREMENTS

Crystal Temperature:	40°C
Ambient Temperature:	0°C - 40°C
Humidity:	Fieldable

D. PMT REQUIREMENTS

Operating Voltage:	1000 VDC

E. LASER SPECIFICATIONS

Wavelengths:	YAG - 532 nm
	Doubled YAG - 266 nm
	Dye Laser - 560 nm - 600 nm (Rhodamine 590 in methanol)
	Doubled Dye Laser - 280 nm - 300 nm
	Optimum Dye Laser - 580 nm
	Optimum Doubled Dye Laser - 290 nm
UV energy output:	>50µJ @ 290 nm

F. DIGITAL OSCILLOSCOPE

PMT Signal:
Vertical:	50 mV/div
Horizontal:	50 nsec/div
Time Base:	Delayed
Time Delay:	450 nsec @ 50m cable
Termination:	50Ω
Vertical Cursors:	Δ = 125 nsec

UV Energy Signal:
Vertical:	50 mV/div
Horizontal:	500 μsec/div
Time Base:	Main
Termination:	1 MΩ
Vertical Cursors:	Δ = 1.25 msec

G. TYPICAL EXPERIMENTAL PARAMETERS

FVD:

Average: 50 shots

Excitation Wavelength:

BTEX - 266 nm

PAH's - 290 nm

Emission Wavelength: Determine for each site

Gasoline - 360 nm

Aviation/Kerosene - 340 nm

Diesel - 360 nm - 400 nm

Coal Tar - 400 nm - 450 nm

Monochromator Slit Width - 200μ - 400μ

WTM:

Average: 200 shots

Excitation Wavelength:

BTEX - 266 nm

PAH's - 290 nm

Emission Wavelengths:

Excitation = 266 nm: 288 nm - 488 nm

Excitation < 300 nm: 320 nm - 500 nm

Wavelength Increment: 10 nm

Monochromator Slit Width - 200μ - 400μ

SECTION V
PRACTICAL OPERATION DETAILS

A. DIGITAL OSCILLOSCOPE

This scope can digitize incoming waveforms at 2 Gigasamples/second. The scope is triggered by a voltage pulse from a photodiode positioned in front of one of the 532 nm reflectors. This triggers the scope with respect to the pump laser. The TDS 620A converts the pulse of electrical current from the PMT into a pulse of voltage and stores it in memory as a digital voltage versus time. Successive pulses can be averaged. Any number of pulses can be averaged, with 50 pulses being the standard averaging rate. DTI recommends a scale of 50 mv/div vertical and 50 ns/div horizontal. The input impedance is 50Ω for the PMT signal (CH2) and photodiode trigger (AUX1) and $1M\Omega$ for the energy meter (CH1). The relatively slow response of the energy meter requires that the time base be set to main only when examining the energy waveform. The peak voltage of the energy waveform from the energy meter must be located between the two time cursors for the software to operate correctly because the software acquires only the data located between the time cursors. The time cursors themselves must be moved to accomplish this. Care must be taken to choose a consistent window of time (usually three divisions to keep data size manageable) for convenient data display and analysis. When monitoring the fluorescence, the scope is run in the *delayed only* time base to allow for a delay between triggering and the return of the fluorescence photons which can be 450 ns for a 50 meter fiber cable. The section of the waveform stored during an experiment is chosen by changing the time delay on the *delay only* time base. Changing the delay has the effect of shifting the waveform back and forth between the two time cursors. Locating the correct timeshift is most easily accomplished by moving the monochromator just off of the excitation wavelength using 12.5 micron slit widths with the fiber-optic cable in place in front of the cone window. Jog the monochromator with the F1 and F2 keys to achieve a 100 mv peak signal. Then change the delay time until the left edge of the laser pulse is ~25ns from the left time cursor. This distance allows complete data collection of the visible wavelengths which arrive sooner in time than the UV wavelengths when a WTM is run. This occurs because of the increase in the speed of light through the fiber with increasing wavelength.

The 532 nm second harmonic output of the YAG laser is the pump laser. This laser directly energizes the dye laser by driving some of the dye molecules in the Bethune prisms into an electronically excited state creating the active lasing medium. The energy of the dye laser is therefore directly influenced by the energy of the YAG laser. The 532 nm light of the YAG can

also be doubled to 266 directly for BTEX capability. Read the Big-Sky manual for details on operation.

B. DYE LASER

The dye laser consists of the two Bethune cells, dye circulator, rotation stage, grating, feedback mirror, and feedback wedge. The dye laser is "angle" tuned by rotating the feedback mirror about the grating. The oscillator cavity consists of the optics located between the feedback mirror and feedback wedge. Amplification is achieved by passing the tuned beam through the amplifier Bethune cell. Thirty percent of the YAG beam is directed into the oscillator cavity and the remainder is directed into the amplifier. The optimum dye laser output wavelength is 580 nm if Rhodamine 590 dye is used. Naphthalene absorbs sufficiently at 290 nm and this wavelength is effectively transmitted by the typical 50 meter SCAPS type fiber optics.

To calibrate the laser, the following steps must be followed while observing safe laser practice. The following procedure requires that the dye laser is properly aligned. This requires hands-on training by DTI. Do not attempt alignment without training!

1. Put a mirror or other shiny nonfluorescent surface in front of fiber probe in a position to reflect a portion of the beam back into the collection fiber. This allows a small amount of the visible light that isn't blocked by the black glass filter to be coupled into the monochromator.

2. Make sure dye laser is lasing. Hit the stepper motor reset button and rotate dye laser rotation stage gear by hand until orange lasing is observed. Again, this step requires that the technician has had some hands-on training by DTI personnel.

3. Jog the monochromator from 560-600 nm while watching for the laser signal on the DSO. Find the wavelength where the signal "peaks out".

4. Call up the Position Laser window (F2). Hit F3 (calibrate) from the Laser Position window and enter the wavelength in its doubled format, i.e. 1/2 the visible wavelength determined with the monochromator. Escape after entering the position.

5. Place a white business card between the launching lens and the black glass filter. Rotate the crystal starting from the position where the visible beam just clears the edges of the crystal. The doubling crystal is rotated by spinning the gear on the rotation stage. Once a blue spot appears, rotate the crystal back and forth until the blue light on the card is maximized. Once this position is found you should go to the Position Crystal window and F3 (calibrate). Hit enter for automatic calibration. Hit escape to exit Position Crystal window.

6. Exit the CS-3 control program and restart CS-3.exe from DOS. Immediately optimize the doubling crystal (F6). The dye laser and doubling crystal are now calibrated.

C. MONOCHROMATOR

The CS-3 software interrogates the 270M as to whether it has been calibrated since it was last powered-up and commands the 270M to undergo a self-calibration if it has not. The wavelength and slit position are stored between each usage and are restored when the system is powered up. Just outside of the exit slit are two slide selectable laser blocking filters. One is a 280 nm cut-off longpass (CFLP) filter which has an OD of 3 at 266 nm and a transmission of > 90% past 285 nm. The other filter is a 320 nm CFLP with an OD of 3 at 290 nm and a transmission > 90% past 320 nm. These filters greatly reduce excitation laser scatter into the PMT (photomultiplier tube). Use the 280 nm CFLP for 266 nm excitation and the 320 nm CFLP for dye laser excitation. One or the other of these filters are always in place. Select the 280 nm CFLP by pulling up on the brass handle at the center of the breadboard under the Plexiglas cover. The 320 CFLP is in place when the handle is down. *(Remember to have the laser turned off when accessing the knob).*

D. PHOTOMULTIPLIER TUBE, HOUSING

The system converts photons to an electrical current with a Hamamatsu® R928 photomultiplier tube in a Products for Research® housing. *Caution!! The PMT high voltage supply must be turned off if the PMT housing or the monochromator are opened for any reason. Permanent damage to the PMT could result from exposure to ambient light levels with high voltage applied!!*

E. FREQUENCY DOUBLING CRYSTAL

The crystal is held at a constant temperature of 40°C by the crystal heater controller to nearly eliminate the effects ambient temperature changes. The crystal temperature can be checked (with laser off!) by looking at the digital readout at the top of the controller. The setpoint is the lower of the two digital numbers. No changes should be made to the set point or other parameters of the crystal temperature controller. An energy meter is used to monitor the doubled beam energy at all times. The CS-3 software is capable of automatically optimize the tuning angle for efficient frequency conversion by running Utilities-Optimize Doubling (F6) in the software. This automatic doubling angle optimization only works when the system currently is producing some doubled light (i.e. is near the optimum angle). It is an optimization technique only. If no doubling is occurring then the Utilities-Locate Doubling feature of the software is run. If dye laser is emitting > 5 mJ energy and the correct doubling angle is not found with the Utilities-Locate Doubling then hand tuning is necessary. Consult the Dye Laser portion of this section for instructions on hand tuning.

F. STEPPER MOTOR CONTROLLER

A Motion Control® stepper motor controller and driver system serves as the control system for the stepper motors located on each of the rotation stages on the breadboard. It is important that the motors are not disconnected from the stepper drivers when power is supplied to them! A 30 second wait is required before disconnecting the motors after powering down. This includes the two 1.5 inch diameter round black connectors on the back of the laser bulkhead. Permanent damage will result if they are disconnected while the power is on. Another danger is supplying a voltage of greater than 120V to the stepper motor system, i.e. the entire cabinet. This causes a varistor to overheat and fail inside the stepper drivers. The stepper motor controller system includes limit switches on the rotation stages to prevent mechanical damage in case of a software failure.

G. POWER REQUIREMENTS

A power source of 30 amp @ 110V is required for the laser cabinet and a 20 amp @ 110V is required for the computer rack. *Note: Voltage must remain under 120V to avoid damaging the stepper motor power supplies!* If generator power is used a 5000W generator is sufficient. The generator must not be allowed to run out of fuel during operation. Damage to electronics could

result. It is important that both racks run off of the same power supply to avoid problems with grounding differences affecting the PMT voltage levels.

H. OPTICS

Laser optics must be kept clean. All optics should be maintained by someone familiar with cleaning fragile optical surfaces. Failure to maintain clean operating conditions will result in premature and expensive component failure.

SECTION VI
EXPERIMENT CONTROL SOFTWARE

The CS-3 software has a pull-down menu system combined with pop-up windows for text and numerical input. The software can be mouse-controlled but DTI recommends keyboard control, as this is the most fully debugged mode of operation. Items at the top of the main menu are selected by first depressing the *alt* key and then "arrowing and entering" or striking the highlighted letters on the menu items.

Experiment setup files avoid the need to tediously input experimental parameters before acquisition of FVDs and WTMs. The file extensions for FVD and WTM experiment setup files are FVX and EMX, respectively.

The menus and windows of the CS-3 software are shown in Figures 2 and 3. A brief description of their purpose and features follows.

A. Main Menu

The main menu screen displays current position of peripherals, current experimental setup choices, and laser energy data. The main menu is the point from which the operator runs experiments and changes settings of peripherals. The software returns to the main menu from all experiment and positioning routines. The DEFAULT.EXX file, displayed under the word EXPERIMENT on the front panel, is an item which is not utilized with this software version and can be ignored. (It is used for excitation spectrum experiments.)

B. *File* Menu

Exit terminates the program. Always exit the program this way.
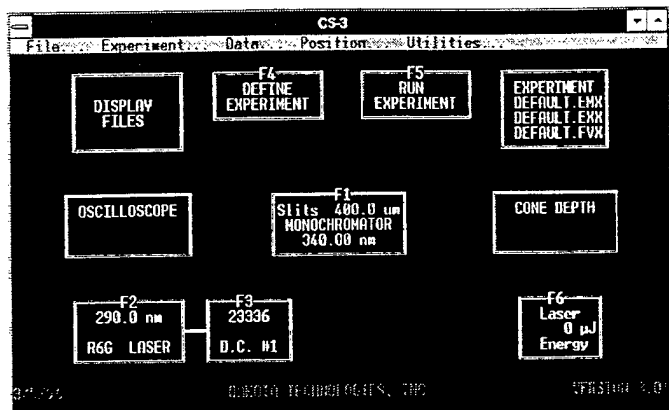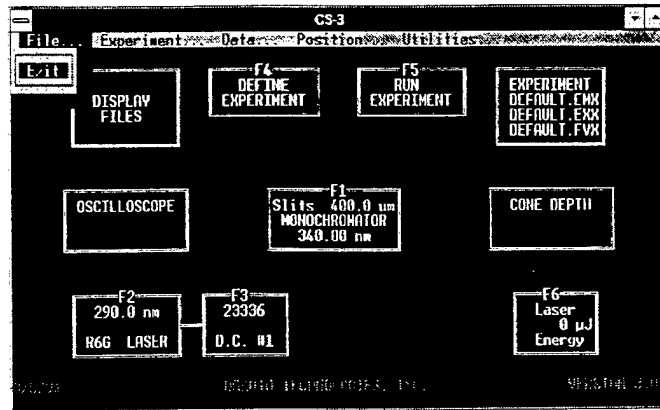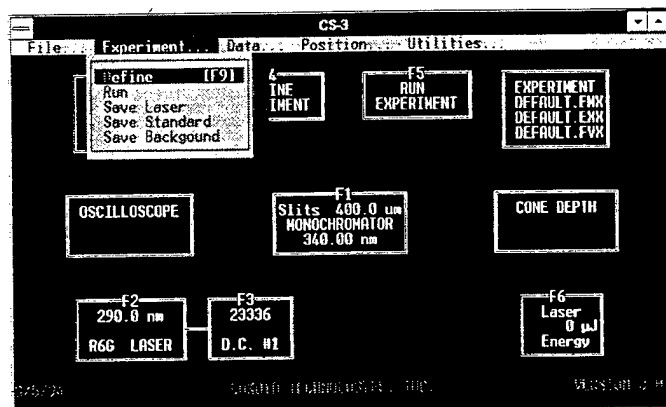
Figure 2.   Pull-down Menus Available in CS-3 Software.
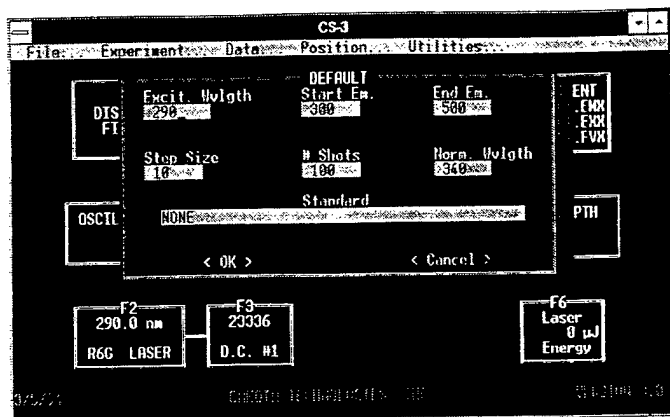
WTM Setup Menu

FVD Setup Menu

Monochromator Position Menu

Dye Laser Position Menu
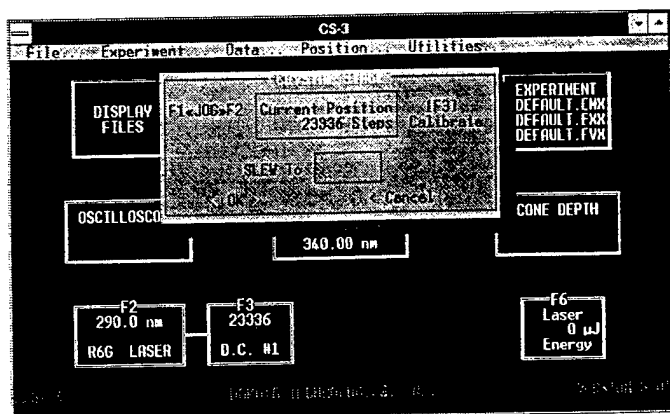
Crystal Position Menu

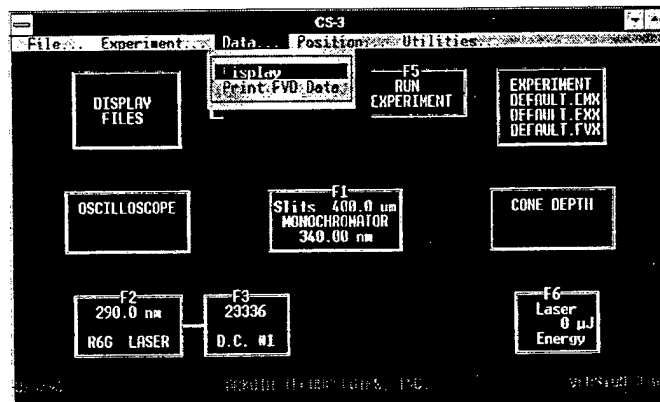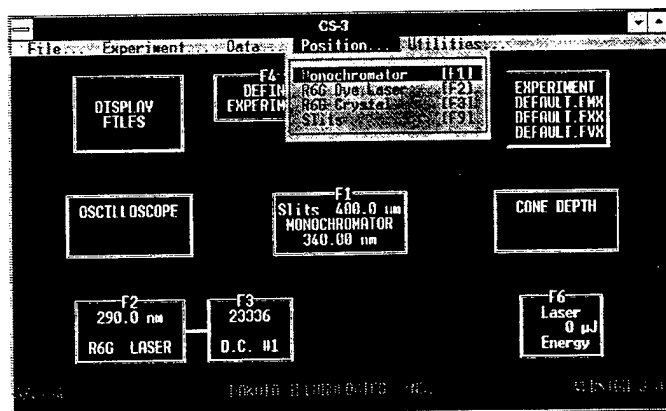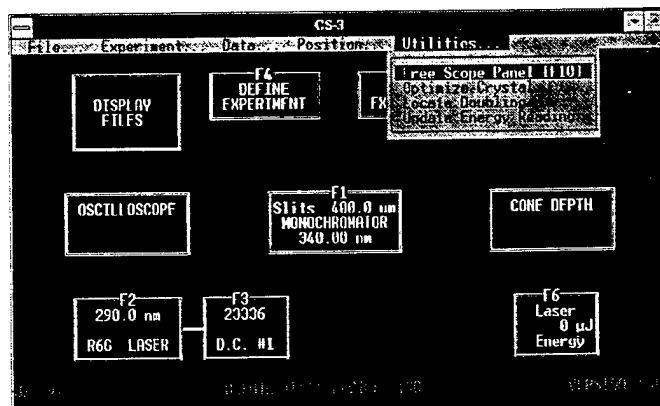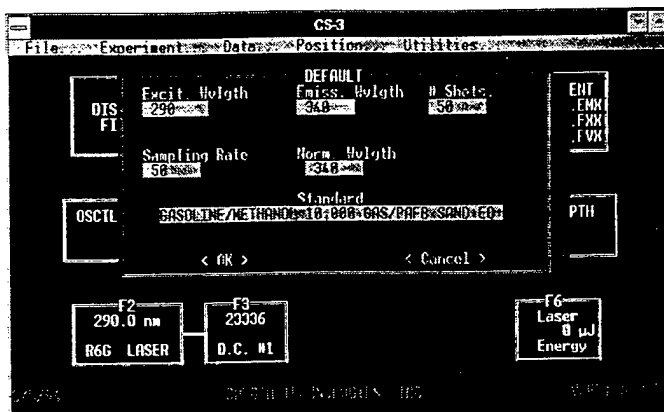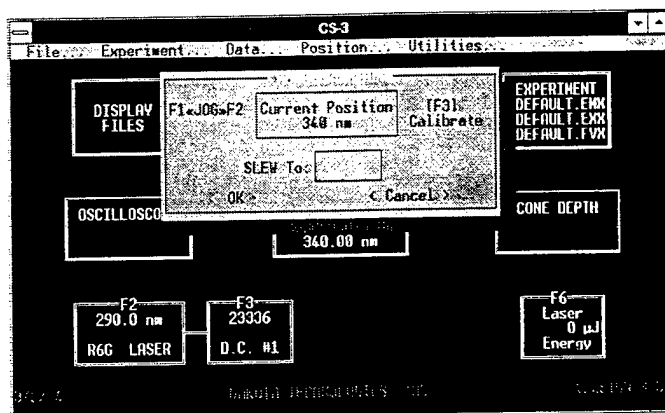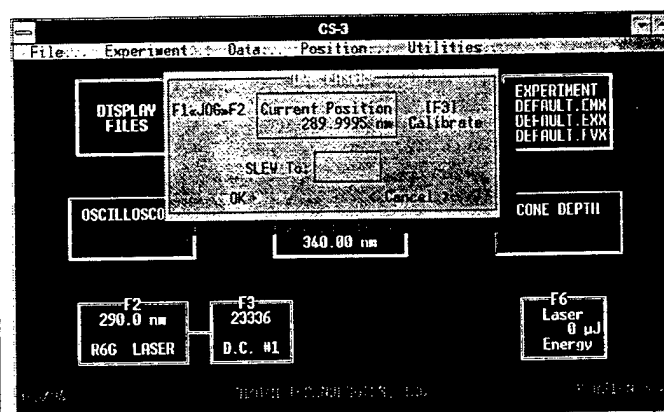Figure 3. Pop-up Window Menus Available in CS-3 Software.

20

C. *Experiment* Menu

1. *Define*

This item allows editing of the wavelength-time-matrix (WTM) or fluorescence versus depth (FVD) experimental setups. Choose the default *c:\bc7\src*.emx* to pick up an emission setup. Backspace and change the *emx* to an *tvx* to pick an FVD from the list.

The WTM setup window is shown in Figure 3. The operator sets the excitation wavelength (laser position), the starting emission wavelength (monochromator position), the ending emission wavelength, the step size (distance moved between waveform acquisitions), the number of shots to average waveform (typically 100-200 shots), the normalization wavelength (monochromator position when the reference solution waveform was stored), and the standard remark (describing the reference solutions composition). These parameters are saved in the HDR (header) extension file of the same name as that of the WTM (stored with a 3DE extension).

The FVD setup is shown in Figure 3. The operator sets the excitation wavelength (laser position), the emission wavelength to monitor during the push, the number of shots to average before downloading each waveform during the push (usually 50, so data are acquired every inch), the sampling rate (disabled in this version of software- do not change default value displayed), the normalization wavelength (monochromator position when the reference solution waveform was stored), and the standard remark (describing the reference solutions composition). These parameters are saved in the PID (push ID) extension file of the same name as that of the FVD (stored with an FVD extension).

2. *Run*

This item is chosen when the operator wishes to execute a WTM or FVD experiment. A window pops up and the operator chooses either an emission experiment (WTM) by striking the *E* key, or a CPT-LIF experiment (FVD) by striking the *F* key. The program then executes one of the two experiments described later in the Section 5.

3. *Save Laser*

This item is chosen when the laser profile is being observed on the

oscilloscope and the operator chooses to save this profile for later use in chemometric calculations on the data. This is very important for lifetime determination. The software averages 500 shots of the signal which is in the window on the scope. It is up to the operator to adjust the monochromator position to establish an acceptable laser signal before saving the laser. DTI recommends saving the laser once a day.

4. *Save Standard*

This item is chosen when the reference solution profile is being observed on the oscilloscope and the operator chooses to save this profile for relating the signal intensity of an FVD to the reference solution. It is very important for quantitative normalization. The software averages 500 shots of the signal which is in the window on the scope. It is up to the operator to make sure that the monochromator position is the same as that to be monitored during the FVD and that the signal is at an acceptable level before saving standard profile. The standard must be saved before every FVD. The standard measurement and acquisition process provides the operator with an excellent opportunity to do a "system check" since the standard signal is a function of all components involved in the FVD measurements, including the downhole optics.

5. *Save Background* (misspelled *Backgound* in code)

This item is chosen when the operator wishes to save the DC background and RF pickup in the coaxial cable and other detector circuitry. This waveform is subtracted from the fluorescence waveforms during the FVD and partially corrects the data for DC and RF pickup. The DC offset drift is somehow affected by temperature and this drift can occur rapidly and can change during an FVD. This prevents a perfect background correction under some conditions. Post processing can correct the drift that occurs during an FVD. The collection fiber must be disconnected during background acquisition or you will not be measuring background! Frequency of this measurement during operation is dependent on temperature stability and the location of the instrument in the CPT truck. These conditions were extremely variable during filed demos of CS-3. Under climate controlled conditions, a morning and noon acquisition might be sufficient.

D. *Data* Menu

1. Display

This item is chosen when the operator wants to display the data after an FVD or WTM has been executed, or to observe past data. Choose *c:\bc7\3de*.3de* to pick from the list of WTM data files in the c:\bc\3de directory. Change *c:\bc7\3de*.3de* to *c:\bc7\3de*.fvd* to display the FVD file list. Once a data set is graphed, the operator can scroll through the waveforms by using the left and right arrow keys. The spectrum in the WTM display and the FVD in the FVD display can be viewed with respect to different delay times by scrolling back and forth

in time with the up and down arrow keys. If a graphics driver is in memory, a screen dump can be made to the printer with the *shift-print screen* key sequence.

2. Print FVD Data

This item is chosen to graph the FVD for printout to a printer. The FVD is displayed with the intensity of the LIF displayed as a percent of the reference solution [(the area of push waveforms/area of reference waveform) x 100]. The print quality is low and this menu option has caused unpredictable results with memory, and only works when data are collected with the scope set at the 50 ns/division time scale. Use with caution.

E. *Position* Menu

1. Monochromator

This item is chosen to change the position of the monochromator. Enter a wavelength in the data entry form labeled "SLEW TO". The *F1* and *F2* keys are used to jog the monochromator small distances in order to locate peak signals (when calibrating for wavelength), or to jog off the peak and get on a "shoulder" of the laser signal for *Save Laser* acquisitions. The user does not need to use the [F3] Calibrate function and should not do so.

2. R6G Dye Laser

This item is chosen to position the rhodamine 6G dye laser. There is limited benefit in changing excitation wavelength from 290 nm. This is where the dye laser operates most efficiently and fiber attenuation is relatively low. If you should want to move the dye laser, you enter a wavelength between 282 and 300 nm. The crystal automatically positions to the correct angle for that wavelength after the laser moves. You should optimize the doubling angle after repositioning the dye laser. The *F1* and *F2* keys jog the dye laser in small increments for fine movement. This is not recommended during normal operation because of the propensity for the operator to press the jog keys faster than the system can respond, sometimes causing an error to occur. The [F3] Calibrate is used to reset the computer's perspective of the position of the dye laser. After checking the actual visible wavelength with the monochromator, the operator uses *F3* in the dye laser positioning window to enter the true wavelength in doubled format. In other words, if the laser is found to be positioned at 593 nm visible, the user strikes the *F3* key while in the R6G Dye Laser position window and enters 296.5 (593.5/2) in the "Current Wavelength

(Doubled!)" data entry form. <u>The frequency doubled laser beam is half the wavelength of the visible dye laser and is displayed at its doubled wavelength in the main menu.</u>

3. R6G Crystal

This item is chosen to position the frequency doubling crystal for the R6G dye laser. The crystal should only be positioned with this item by technicians working on the dye laser-doubling tracking algorithms. There is no need for the operator to use this item under normal operating conditions. The [F3] Calibrate is used when the crystal is hand-tuned into doubling range when the "Locate Doubling" and "Optimize Crystal" have failed to locate the correct frequency-doubling angle. When doubling is achieved the operator strikes the *Enter* key for "Autocalibration", then escapes out of the positioning window. The autocalibration simply tells the computer to calculate the expected position for the doubling angle at the present dye laser position using the positioning algorithm, then the computer stores that position as the current crystal position.

4. Slits

This item brings up a data entry window in which the operator enters the slit width in microns for both the entry and exit slits on the monochromator. The slits should not be opened past 1000 microns. The typical slit width for FVDs and WTMs is between 100-600 microns. Slits affect the bandwidth of the monochromator. Narrow slits (12 microns) should be used for laser calibration. Use the slit width to achieve proper signal levels for the reference solution. Wider slits let more light (wider bandpass) into the PMT.

F. *Utilities* Menu

1. Free Scope Panel

This item is chosen to release the scope from a panel lockout state which occasionally occurs. If the scope does not respond to its front panel, simply choose this menu item to free it.

2. Optimize Crystal

This item causes the crystal to be moved through a series of angles about the current doubling angle. The doubled dye laser energy is monitored by measuring the intensity of an electrical pulse from the Molectron® energy meter connected to CH1 on the scope during rotation of the crystal through this series of angles. The angle which achieves the best efficiency is returned to and a final measurement of laser energy is made and displayed on the front panel. Although the crystal temperature is held steady by a heater/controller, drastic changes in ambient operating environment temperatures (30° F in the morning to 80°F by noon) cause the crystal temperature to drift. Use this item to occasionally optimize doubling efficiency. This is a "fine tune" procedure.

3. Locate Doubling

This item is a coarser version of Optimize Crystal. It is used if "Optimize Crystal" fails to go through a maximum and find efficient doubling. Use this only once and only when satisfactory visible dye laser performance (>5 mJ/pulse energy) has been confirmed by a safety-trained laser technician. If used when dye laser performance is poor, the crystal will get "lost" and will have to be hand-tuned back into position. Remember to "Autocalibrate" crystal position when hand-tuning.

4. Update Energy Reading

This item is chosen when the operator wants to take an energy reading of the doubled dye laser energy without optimizing the doubling angle. This is faster than the "Optimize Crystal" and allows the operator to get an energy reading after an FVD without changing the angle from what it was during the FVD . This is useful for documenting laser energy drift during FVDs.

SECTION VII
DATA FILE FORMAT DESCRIPTIONS

A. FVD EXPERIMENTAL FILES

Six separate data files are created at the successful completion of an FVD. Each file has the same name as the FVD file, but a different extension. The extensions are listed below with a description of the file formats.

1. FVD Extension (Fluorescence Versus Depth)

This file stores all of the waveforms acquired during an FVD push. The data are stored as a space-delimited matrix with carriage return characters (ASCII character 13) at the end of each row. A zero in the upper left corner (first data point in file) serves as a place holder for the empty location outside of the matrix. The rest of the top row is the time vector. This represents the window in time between the cursors of the scope during the experiment. The time is stored in units of nanoseconds. In column 1 of the FVD is the depth below the ground surface of the cone tip (not the window) in units of feet. After each depth is a row of data points which represents the waveform at that depth stored in units of volts.

2. PID Extension (Push ID)

This file contains all of the information about the FVD. The first row contains the excitation wavelength used (in nm), the emission wavelength (in nm), the number of waveforms saved (number of discrete depths), the number of shots averaged between waveform downloads, the frequency value (obsolete), the normalization wavelength, and the reference solution comment, respectively. The second row contains the width of the time window in units of ns. The third row contains the time between voltage data points in units of seconds. The fourth row contains the header from the scope. This stores all scope setup information at the time of push completion. Consult DSO manuals for details. The fifth row contains the current time at the completion push according to the PC's clock. The sixth row contains the current date according to the PC's clock. The seventh row contains the energy of the dye laser from the last time the energy was measured by the control computer. The eighth row contains the area of the reference solution in pico-volt-seconds which is calculated by the DSO.

3. PLS Extension (Push laser)

The laser waveform which was saved in the scope memory the last time the laser profile was saved. Column 1 is time in ns. Column 2 is volts.

4. PST Extension (Push standard)

The reference solution waveform which was saved in the scope memory the last time the reference profile was saved. Column 1 is time in ns. Column 2 is volts.

5. BKD Extension (Background)

The background waveform which was saved in the scope memory the last time the background profile was saved. Column 1 is time in ns. Column 2 is volts.

6. AVD Extension (Area vs Depth)

This is a very simple way of looking at the results of a push. This file contains the window offset corrected depth in column 1 (the actual window depth). Column 2 contains the area under the curve of the fluorescence waveform acquired at the matching depth in column 1. Units are in pico-volt-seconds. The *Data-Print FVD Data* feature of the software uses this file to print out the FVD in % of reference solution. DTI suggests that the FVD file, along with the PST and BKD files, be used to calculate and graph LIF response figures. The AVD file and the *Data-Print FVD Data* feature of the software are meant to be used for rudimentary field viewing only!

B. WTM Experimental Files

Four different data files are created every time a WTM is successfully completed. The name you choose for the WTM file is also used as the name for three other files. The extensions are listed below with a description of the file formats.

1. 3DE Extension (3-Dimensional Emission)

This file stores all of the waveforms acquired during WTM push. The data are stored as a space delimited matrix with carriage return characters (ASCII character 13) at the

end of each row. In the upper left corner (first data point in file) is a zero. This is simply a place holder for the empty location outside of the matrix. The rest of the top row is the time vector. This represents the window in time between the cursors of the oscilloscope during the experiment. The time is stored in units of nanoseconds. Column 1 is the wavelength of the window in units of feet. After each depth is a row of data points which represent the waveform at that depth stored in units of volts.

2. HDR Extension (Header)

This file contains all of the information about the WTM. The first row contains the excitation wavelength used (nm), the starting emission wavelength (nm), the ending emission wavelength (nm), the step size (increment) in nm, the number of shots averaged at each wavelength, the normalization wavelength, and the reference solution comment, respectively. The second row contains the width of the time window in units of ns. The third row contains the header from the scope. This stores all scope setup information at the time of WTM completion. Consult oscilloscope manuals for details. The fourth row contains the current time at the completion of the push according to the PC's clock. The fifth row contains the current date according to the PC's clock. The sixth row contains the current probe tip depth in feet. The seventh row contains the energy of the dye laser from the last time the energy was measured by the control computer. The eighth row contains the area of the reference solution in pico-volt-seconds which is calculated by the DSO. The integrity of this data is questionable - do not rely on this value; calculate the area from the STD file.

3. LSR Extension (Laser)

The laser waveform which was saved in the scopes memory the last time the laser profile was saved. Column 1 is time in ns. Column 2 is volts.

4. STD Extension (Standard)

The reference solution waveform which was saved in the scope's memory the last time the reference profile was saved. Column 1 is time in ns. Column 2 is volts.

SECTION VIII
EXPERIMENTAL SEQUENCE DESCRIPTIONS

At this point it is appropriate to walk through the sequence of events for a single fluorescence measurement and both the WTM and FVD experimental modes of CS-3. This will aid the operator in grasping the "big picture" of the CS-3 system.

A. A SINGLE FLUORESCENCE MEASUREMENT

A long chain of events occurs each time the laser fires. The following describes what happens 50 times every second with the CS-3 system.

ᖶ The YAG laser fires and a 12ns wide pulse of 532 nm light exits the front port of the laser head.

ᖶ The YAG beam strikes one and then another 100% reflective mirror and is now headed straight toward the oscillator bethune prism. A very small portion of the YAG beam is scattered randomly by dust, etc. on the mirrors and some of this light strikes a photodiode. The photons striking the photodiode are converted into a pulse of current which is carried to the DSO in a coaxial cable. This current creates a pulse of voltage at the DSO and this triggers the DSO to begin data conversion. If the scope is in the delayed mode, the scope begins to keep track of the amount of time passed and will begin conversion at the end of the delay.

ᖶ The YAG beam passes through a 70% reflective beam-splitter. 70% of the beam is turned 90° and is headed toward the amplifier bethune cell.

ᖶ The remaining 30% enters the oscillator bethune cell. The YAG beam begins to be absorbed by the dye in the bore of the cell.

ᖶ The dye begins to emit fluorescence in all directions. Some of the emission occurs along the axis of the bore and strikes remaining excited state dye molecules which spontaneously emit two photons of the same wavelength of the photon which was absorbed. This creates a diffuse but intense beam coming out both ends of the bore.

30

⇲   Some of the light emitted from the bore strikes a wedge prism next to the cell. It is positioned at just the right angle to reflect ~4% of the light directly back through the bore of the cell. This light strikes more excited state dye molecules and spontaneous emission "cascades" through the bore creating a flux of broadband light which passes through the cell to the other side.

⇲   This beam of wideband fluorescence strikes a grating at a slight angle ("grazing incidence"). A small amount of this beam undergoes first order diffraction from the grating and is dispersed at varying angles according to the wavelength (i.e. energy) of each photon in the beam. Most of the beam simply reflects off of the grating and is lost.

⇲   A mirror attached to the rotation stage reflects a very narrow range of wavelengths from this refracted light directly back to the grating where it is again diffracted back into the bore, where more excited state dye molecules are being created by the YAG beam. These "selected" photons sweep back through the bore and create a "cascade" of photons of the "selected" wavelength. The wavelength of the selected light is controlled by the angle of the feedback mirror. This angle is controlled by moving the rotation stage. This cycle of photons sweeping back and forth through the bore is what creates the tunable laser beam. Remember that only 4% of the beam is reflected back through the cell by the wedge prism. The remaining 92% passed through (4% is lost off the 2nd wedge surface) and these photons are headed along the axis of the bore of the amplifier cell toward it.

⇲   At this point the 70% of the YAG beam that remained is entering the amplifier. As the oscillator beam passes through the bethune cell it collides with a "cylinder" of excited state dye molecules created by the arriving YAG beam and undergoes an increase in energy. This is why this cell is called the amplifier. Its sole function is to intensify the beam which has been "tuned" to the desired wavelength in the oscillator cavity.

⇲   The dye laser beam exits the amplifier at full intensity, enters a turning prism, and is directed into the doubling crystal.

⇲   As the laser beam travels through the doubling crystal it is converted into a laser beam of twice the frequency, or half the wavelength, of the visible beam. This

happens at a very precise angle and temperature. These parameters are controlled by the rotation stage and crystal temperature controller respectively.

♨ The beam exits the crystal and strikes a visible blocking filter which is positioned at ~40° to the beam. A fraction of the combined visible/UV beam is reflected toward and strikes a joulemeter with a visible blocking filter positioned in front of the sensor element. This element converts the photons into an electrical current pulse which is carried to the DSO by a coaxial cable where it can be observed visually and monitored by the computer.

♨ Most of the beam is absorbed by the visible blocking filter as it attempts to pass through. A very small amount of visible energy passes through because the filter is not totally opaque to the visible wavelengths, especially in the green zone of the visible spectrum.

♨ Exiting the filter is a nearly pure UV beam of the selected wavelength. It passes through a 40mm focal length bi-convex convex silica lens and is focused into the fiberoptic.

♨ The light now travels through the fiber, continuously being reflected off the wall of the fiber because of total internal reflection. The UV beam exits the end of the fiber, goes through the sapphire window, and strikes the matrix (soil, water, etc.) being analyzed.

At this point several things can happen.

1. Laser light reflects off of the matrix and some portion enters the collection fiber to be carried back to CS-3.

2. If contamination is present, laser light is absorbed by the molecules in the matrix (petroleums, oils, lubricants (POLs), etc.) driving them into an electronically excited state. These molecules can get back to the ground state by collision with neighboring molecules, phosphorescence, fluorescence, etc. It is the fluorescence photons that we are interested in. Some molecules wait much longer than others to emit their fluorescence photons. Therefore, photons are

32

being emitted over a range of time, from fractions of nanoseconds to hundreds of nanoseconds after the laser pulse, depending on the contaminant matrix and the contaminant molecules present. Some of the fluorescence will couple back into the collection fiber.

3.   Laser light undergoes Raman scattering by molecules in the matrix. The intensity of these scattered photons is too low to concern us in comparison to the fluorescence.

4.   Laser light is simply absorbed by the matrix and is converted into heat.

↻   A pulse of photons are now headed toward the monochromator. Some are reflected laser light and some are fluorescence.

↻   The photons exit the collection fiber and are directed toward the entrance slit on the monochromator. If the slit is wider than the fiber, all the photons enter the monochromator. Some are rejected by the slit if the slit is narrower than the fiber. Slit width determines the range of wavelengths or bandwidth of photons which will make it through the monochromator.

↻   The photons are diverging as they head toward the first mirror in the monochromator. They strike the mirror and are reflected toward the grating. At this point they are nearly collimated by the reflection off the concave spherical mirror.

↻   The photons strike the grating and are diffracted at varying angles according to their wavelength. This creates a rainbow distribution headed toward the second concave spherical mirror. This second mirror refocuses the photons into a slit shape that is directed toward the exit slit. The image on the exit aperture is a horizontally dispersed rainbow of colors (different wavelength photons). The angle of the grating controls which narrow range of the rainbow is positioned on the exit slit.

↻   The range of photons desired (controlled by software selection of a position for the monochromator) passes through the exit slit and strikes the laser filter. Monochromators are not perfect devices and some laser photons invariably make it through the monochromator. The laser filter absorbs and/or reflects the most of the

laser wavelength photons which made it through the slits and lets the fluorescence photons through.

☞ The fluorescence photons are "strung out" in space at this time. Some are through the monochromator and begin striking the photomultiplier tube (PMT) while some are still traveling through the fiber.

☞ As the photons arrive they hit the photocathode in the PMT. The photocathode is made of a material that ejects electrons when photons are absorbed. A chain of dynodes under a high voltage bias (900-1200V) is located in close proximity to the photocathode. When electrons are ejected they are pulled toward the dynode chain. The electrons jump from dynode to dynode. With each dynode there is an increase in the number of electrons which are supplied by the high voltage bias power supply. This causes a "cascade" of electrons for each photon which strike the photocathode. This is why the PMT is known as an amplifying device, i.e. many electrons are available to measure for each photon striking the PMT, resulting in a very sensitive photon measuring device. The net effect is that the train of photons are converted into a pulse of electrical current. This current is carried to the DSO with a coaxial cable for measurement.

☞ The electrical pulse arrives at the DSO where a 50Ω terminator converts the current to a voltage. The oscilloscope has been waiting the desired amount of time and just before the pulse arrives it has begun to very quickly measure the voltage vs time. One 12 bit voltage measurement every 0.5 or 1.0ns is typical for CS-3.

☞ The waveform (the voltage vs time data set) is stored in the scopes memory. The computer can now access this waveform via the GPIB interface.

And thus one fluorescence measurement has taken place in 20 microseconds! Typically 50 to 200 of these waveforms will be averaged (to a 16 bit result) by the DSO before the computer downloads the waveform.

B. FVD EXPERIMENT

We start with lasers running at proper energy level. This is checked with the dye laser energy meter.

꩜   EMX and FVX experimental file parameters are set to desired settings.

꩜   Monochromator is positioned at emission wavelength which will be the best indicator of POL contamination. The wavelength chosen should offer sensitivity to the contaminant and resistance to naturally occurring interfering fluorescence (420 nm and longer). 340 and 360 nm are typical. This wavelength is also set as emission wavelength in FVX file.

꩜   Reference solution which mimics contaminant is placed in front of CPT window.

꩜   Laser energy and/or monochromator slit width is adjusted to achieve desired intensity on scope (~100mV peak).

꩜   Reference signal is saved using *alt-Experiment-Save Standard*. This is done to normalize data (to account for laser energy drift, etc.) and to allow operator to have a rough feel for concentration of contaminant sensed.

꩜   Monochromator is positioned near laser wavelength with nothing in front of clean CPT window. The monochromator is jogged to allow laser pulse to be seen on scope at desired intensity (100 mV) on scope.

꩜   Laser signal is saved using *alt-Experiment-Save Laser*. This signal is saved for post-processing of fluorescence lifetimes for chemometrics.

꩜   Collection fiber is disconnected from back of laser cabinet. Background signal is saved to memory using *alt-Experiment-Save Background*. This eliminates the sometimes large "background" shifts seen during soundings.

꩜   Operator types *alt-E-R* and enters *F* for Fluorescence versus Depth (FVD).

꩜   Operator enters sounding file name. Overwrite protection is offered.

꩜   Operator gets signal from CPT operator that the system is at ground level and is ready to proceed with sounding.

꩜   Operator hits enter at comment prompt.

- The computer program sets a trap for the F10 and F12 keys. These are the WTM key and complete push keys respectively. Any time these keys are hit the program will be interrupted and will service these events. These events are discussed later in this series.

- The computer parks all the peripheral devices in the locations defined in the FVX file.

- The computer acquires all the reference waveforms in the scopes memory and sets scope to average the number of shots defined in FVX file.

- The computer queries the scope on the time base. The computer then creates a new appendable file called a *.*FVD* file, with the * being the name chosen by the operator. The computer saves a vector of ASCII time values as the first row in the file. This vector represents the window of time being sampled by the scope.

- The computer resets the scope to zero shots and commands scope to begin new series of averages. The computer begins a constant query of the scope for number of trigger events.

- When number of trigger events equals the desired average, the computer queries the scope for a waveform and, while the scope is answering, updates the cone position by reading the depth in 1/10000 of a foot from the 32 bit I/O board.

- The scope sends the waveform data points as a string of 16 bit numbers. The conversion factors such as Δtime between points, voltage per digital level, DC offset, etc. are also downloaded. The computer converts the digital waveform to analog voltage levels and appends the FVD file with the current tip depth in column 1 and the corresponding waveform data values in the remaining columns of the FVD file, one data point for each point in time across row 1 of the file. In this fashion the depth and waveform are saved in a matrix style table of ASCII characters. Immediate saving of each waveform prevents data loss in case of system failure during the push (power failure, etc.).

- The background waveform area is subtracted from the fluorescence waveform area and the result (in pico-volt-seconds) is sent to the CPT control computer on the I/O card.

- The computer stays in a loop, querying the scope and I/O card, sending data to the CPT computer, and saving data every time the scope has averaged the desired number of shots. Data are saved only when the depth has increased from the last depth saved. This prevents huge data files from being created during long pauses in penetration.

- When the operator sees an interesting zone of contamination he may desire to know more about the spectral properties of the fluorescence being emitted by the contaminant. He tells the operator to pause the push and strikes the F10 key to run a WTM.

- The computer saves the operating parameters being used for the FVD into memory. A menu pops up and allows the operator to choose the desired WTM experimental parameters (EMX file). After the operator chooses the appropriate file and saves any changes, the computer runs a WTM in the following manner.

C. WTM EXPERIMENT

- The computer acquires the reference waveforms from the scope.

- The computer resets the peripherals to the settings chosen in the EMX file.

- The computer slews monochromator to the beginning emission position.

- The scope is commanded to average the desired number of shots (typically 100-200 shots).

- A new file is created called a 3DE file. The first row is again a vector of ASCII time values.

- The scope is queried constantly and when the desired number of shots are taken, the waveform is downloaded and converted just as it is in the FVD mode. After the waveform is converted the current wavelength setting is saved to column one of the 3DE file and the waveform is saved to the remaining columns

↳ This is repeated for all wavelength positions defined in the EMX file. This results in a complete 3-dimensional picture of the fluorescence emission. At the end of the WTM the laser and reference material waveforms are saved in *.lsr and *.std files, respectively. The acquisition parameters, time, date, etc. are saved in a *.hdr file.

↳ The operating parameters of the FVD are restored and the FVD continues.

↳ As many WTMs as desired can be acquired.

↳ When rejection occurs or the desired depth is achieved, the operator strikes the F12 key. This causes the computer to save the laser and reference material waveforms to *.pls and *.pst files, respectively. The operating parameters, time, date, etc. are saved to a *.pid file.

↳ The computer returns to the main menu.

SECTION IX
SYSTEM STARTUP, OPERATION, AND SHUTDOWN

A. STARTUP AND OPERATION

1. Ensure that the communications cables and the coax cables are properly connected to the back of the two cabinets. Connect power source to both racks. Turn on laser rack and computer rack by flipping main switches on upper right corners at the back of each rack. Turn on the oscilloscope and computer. Observe crystal controller through the front view port on the laser. The crystal temperature should be increasing toward the setpoint temperature of 40 degrees Celsius (located at bottom of display). Check water level in the reservoir located in the laser drawer. If water is not visible in the viewer located on the laser drawer, you must add deionized water to the reservoir. Be very careful not to contaminate the water when you are topping off the reservoir. Turn key to ON position on the Big Sky laser controller and wait 10 minutes. Confirm that the crystal temperature in the viewport is within $0.3°$ of setpoint. Press the RUN pad on the Big Sky controller. If a warning concerning oven temperature is seen, then press the CLR pad and wait several minutes. DO NOT PRESS RUN WHEN WARNING IS DISPLAYED! After clearing and waiting, press RUN again and check for warning. When doubling crystal inside the YAG is sufficiently warm the laser will run without the warning indicator coming up. Once laser is running wait 10 seconds and check for triggering of the digital oscilloscope. If no triggering is seen use the shutter control (Item E) on the main menu to open the shutter on the YAG. Once triggering is confirmed, the system should be allowed to run for 15 minutes before proceeding to next step.

2. The control program will boot automatically. If you exit from the control program and you need to restart it, go to directory c:and TYPE "CS3<cr>" Do not run as a shelled program from Windows®, Norton Commander®, etc. The laser must be running for the control program to work.

3. At boot-up, the system will advise you on communication status with the monochromator and laser positioning system. If monochromator communication is not established, the monochromator should be rebooted and the control program should be exited and restarted. Stepper motor communication follows successful monochromator

communication. If stepper communication is not established, a warning is displayed prompting the operator to press the stepper motor reset button located on the upper right corner of the front of the laser cabinet. This should allow communication to take place. If not, check all serial connections and/or seek technical assistance.

4. Doubling crystal optimization is achieved by rotating the crystal through a series of angles and determining and returning the crystal to that angle. Doubling crystal optimization should be attempted only after at least 15 minutes of laser warm-up. Use the *alt-Utilities-Optimize Crystal* command or hit F6 from the front panel of the control program to optimize the crystal. Observe the oscilloscope panel while the crystal is being optimized. There should be a noticeable rise and fall of the waveform on the bottom of the screen. If optimization is not achieved (energy < 40 µJ) and no noticeable rise and fall of the waveform has occurred then exit the control program (alt-*File-Exit*). Double check the crystal temperature controller. It must be within .3° C of the setpoint for the software routines to find the doubling angle! Hit the position controller reset button (red) on the front of the laser rack. Run the CS3 program again. Note whether the stepper communication is established. If so, attempt another crystal optimization. If no improvement is seen then run *alt-Utilities-Locate Doubling*. This routine runs a wider search pattern for the correct doubling angle. If doubling is located then run *alt-Utilities-Optimize Crystal* one or two times to "zero in" on the correct doubling angle. If no doubling is found, exit the program and follow the steps in the next paragraph.

At this point a manual check of the system is necessary. If you are a trained operator, check that both the YAG and dye laser pulse energies are satisfactory (>30 mJ YAG and > 5 mJ dye laser) and the dye laser is positioned at the proper wavelength. If you are not trained in laser safety, you must have someone who is trained service your laser. Under no conditions should an untrained operator attempt laser adjustments with the cover removed!! Make sure you are wearing safety goggles and remove the cover from the laser. Defeat the interlock on the back right bulkhead with the o-ring near the switch. Be sure you have laser goggles on! If the interlock stopped the laser, hit RUN to restart it. While watching the doubling crystal rotation stage ring gear, optimize the doubling crystal with the control program. If the gears on the crystal stage do not turn then the motorized positioning system may be damaged. Until repair is made, it is possible to "hand tune". If you are certain that the visible dye laser is operating correctly, place a bright white business card just in front of the lens (the optical element

just in front of the fiber). Rotate the ring gear back and forth (it will offer resistance if stepper power is on) until you notice a bright blue spot on the card. Rotate slowly to avoid "missing" the angle at which doubling occurs. Optimize angle for best doubling. Don't forget to remove the card before replacing the cover. Once doubling has been computer optimized you can adjust the Lamp Energy (Item B) on the BigSky laser to get more tunable UV energy. Adjust power up in increments of 0.1 joules. The 532 nm beam should be measured with each adjustment (measurable with a power meter not supplied with system. Call DTI for guidance in this matter). Do a crystal optimization between increments to reassess tunable laser energy. Do not exceed 100 μJ of tunable UV energy or 40 mJ of 532nm pump energy. Be very careful when adjusting the BigSky laser. Accidentally entering too high of a lamp energy (> 8.5 J) could permanently damage the dye laser and almost certainly fracture the launch fiber. If you need to go higher than 8.5 Joules energy, please call DTI for advice on dye change or flash lamp replacement.

5. <u>CPT preparation steps</u>. Once the system is operating at satisfactory ultraviolet laser energy (> 40 uJ/pulse) the following steps are suggested prior to initiation of a LIF-CPT push.

     a. Run a crystal angle optimization.

     b. Clean the sapphire window and position the reference solution in front of the window.

     c Verify that the monochromator is set to desired LIF-CPT emission wavelength (usually 340 nm).

     d. Verify that CH2 is the active channel on oscilloscope (CH2 light is on).

     e. Set Acquire-Average to 50 (from the value of 800 used in the crystal optimization).

     f. Use vertical control knob on the oscilloscope to adjust vertical scale to 50mV/division.

g. Adjust the monochromator slits and/or laser energy to obtain a one-third full scale vertical deflection on the oscilloscope. If standard is giving virtually no signal then you should confirm that laser light is exiting the cone with the aid of a business card. If not, you should disconnect the probe and check the connector coming from the laser. If no UV light is coming out of the window but UV light is coming out the rear laser fiber bulkhead on the laser then something is wrong with the light train between the bulkhead and the window. Check optics in cone and fiber optics connector/fibers.

If energy is good according to the CS-3 control program, but no or very little light is coming out of the laser bulkhead, then the launch fiber is misaligned or damaged. Call DTI for assistance in realigning fiber or call fiber supplier for advise on repolishing the launch fiber.

h. Use the *alt-Experiment-Save Standard* command to save the standard.

i. Insure that the proper Fluorescence vs. Depth experimental file (FVX file) hase been selected. If not, load it via the *alt-Experiment-Define* command. Enter *c:.fvx* in place of the *c:.emx* in the pop-up window. Hit ENTER. Select the desired FVX file and hit ENTER. If you wish to customize the set-up you tab through and change the parameters, hit ENTER, and then enter a filename of eight characters or less with no extension. Hit ENTER again. Remember to load your custom configured set-up every time you boot up because CS-3 software loads default.fvx on boot-up.

j. Remove the reference solution and notify the CPT operator that the system is ready. When confirmation is given, use the *alt-Experiment-Run* command and hit the *F* key to run an FVD. A 10 second delay will occur while communication with the truck and scope is being established. When the active push screen comes up, the CPT operator can begin the push.

k. WTMs can be initiated at any time during the push by hitting the F10 key. A pop-up window will prompt you for the WTM experiment filename. Use the C:.EMX given and hit ENTER. Choose the Default or a customized setup and hit ENTER three times to register current setup. The system will acquire a WTM, prompt for a filename, then return to the active push screen when

finished. Any number of WTMs can be run at any point in the push. If a WTM is run at the bottom of a push, it is recommended that a small push be continued after the WTM is acquired before using the F12 key to terminate the push. Hit ESC at the end of the push to return to the main menu. Memory limitations prevent a full viewing of some larger FVD files. Use the freeze screen at the end of the push to view the push. You can re-examine the file from the data collection program using the *alt-Data-Display* subject to memory restrictions.

B. SYSTEM IDLE AND SHUTDOWN

If the system will not be used within the next hour, it is recommended that the laser be STOPped. The flashlamps in the YAG itself are degraded with every flash. Stopping the laser will extend the interval between lamp replacement and the dye degradation due to photodegradation from exposure to the YAG beam. A 15 minute warm-up should be sufficient time for stabilization before each push.

The YAG should be idled (fans are running but laser not firing) for at least 1 minute before turning the key to the off position to allow the lamps to cool. Once the system has shut down (~10 seconds after the key is turned off) the system can be disconnected from power. Exit the CS-3 software and turn the computer and scope off, then disconnect power.

C. REFERENCE SOLUTIONS AND "CALIBRATION"

DTI suggests a single point "calibration" of this instrument (this is how the software is designed) with solutions that are composed of the suspected site contaminant diluted with methanol or an aliphatic solvent (gasoline, JP-4, diesel, coal tar, etc.), even recovered product itself. If the contaminant is unknown, use a diesel standard because of its broad emission. Solutions should yield a waveform area equivalent to the waveform area yielded by a 10,000 ppm mixture of the suspected contaminant on Fisher sand. The slits should be adjusted on the monochromator to yield a signal close to -150 mV baseline to peak voltage on the scope (3 divisions at the suggested 50mV/div vertical setting). This has allowed for linear signal/concentration behavior over several orders of magnitude in laboratory experiments. The waveforms for both the sand and solution mixes should be measured at the wavelength of maximum emission for the suspected contaminant.

Single point calibrations give the operator a "rough feel" for concentration and this is often sufficient for well placement decisions, non-aqueous phase plume delineation, petroleum-based

LNAPL/DNAPL characterization, etc. The single reference solution gives you this capability. It is also a "system check" - any problems with CS-3 can be detected by monitoring the quality and amplitude of the reference solution signal prior to each push.

D. GENERAL WARNINGS

The following is a list of "don'ts" for those not extremely familiar with the system and all of the components.

♦ Do not turn on any "measurements" on the scope and leave them on during a run. The scope is unable to keep up with the demands of the system and do measurements at the same time. Running the system with measurements on will force the system to slow down or give spurious results! If "measurements" are needed, make them and remove the "measurements" before doing CPT experiments.

♦ Avoid changing settings on the scope if you are not experienced and you do not understand the functions of the scope. Past experience has shown that once the scope has been properly set up the operator only needs to set the vertical scaling and run-stop buttons to achieve satisfactory measurements. If you are unsure of the scope settings use the recall-setup function to download "setup1" or "setup2" from the scope memory to "get you in the ballpark".

♦ Do not remove the laser cover without goggles on! You must never put your eyes at table level. This exposes you to risk of eye injury. If you must remove the cover you should be briefed on laser safety first. The Big-Sky manual should be consulted for laser safety.

♦ Never disconnect the large round black electrical connectors (or any others for that matter) on the laser bulkhead with the system powered up! This will cause permanent damage to the stepper motor modules.